WASHINGTON SQUARE WEST, PHILADELPHIA, PA. 19105 TELEPHONE (215) 592-3000

REPLY TO:
BOX 219
BRISTOL, PA. 19007
(215) 788-6501

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Received NOV 24 1970

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ROHM HAAS COMPANY

November 20, 1970

Francis T. Brezenski
United States Dept. of the Interior
Federal Water Pollution Control Admin.
Northeast Region
Hudson-Delaware Basins Office
Edison, New Jersey 08817

Dear Mr. Brezenski;

Confirming our telephone discussion of today, we plan to meet you at your office at 10:00 a.m., December 1 to discuss analytical procedures and methodology. Besides myself, I expect that Mr. John Kauffman, Laboratory Director, Whitmoyer Laboratories, and Messrs. Walter Zook and Herman Behrend, Laboratory Heads from the Bristol Plant to accompany me. Aside from our analysis of arsenic in stream bottom muds, we appreciate your offer to discuss the analysis of other heavy metals as well.

Until then, I wish you and Al a very Happy Thanksgiving.

Sincerely,

ROHM AND HAAS COMPANY

T. Iezzi

TI:mjd
cc: Mr. Ambrogi/Kauffman ✓
Mr. Behrend/Mr. Zook
Mr. Dengler
Dr. Gutbezahl
Dr. Kelton
Mr. Paist
Mr. Rarig
Dr. Winters

Contrary to Mr. Felton's report of his conversation with Mr. Sterling, Corp. of Engineers, that arsenic is no longer a factor in the hold up of construction of the Blue Marsh Dam, Mr. E. Geismar, FWQA, told me at the recent meeting in the Poconos that the Corp. of Engineers is pressing for a decision on the arsenic from his agency. Mr. Geismar said "you can't blame them and we are still trying to get a big meeting together." Setting the above meeting date with Mr. Brezenski was at his request, to resolve differences in analysis of stream bottom mud samples which we are sharing with his laboratory.

AR100163

Copies of recommended Methodology for lead, cadmium and arsenic sent to me by Mr. Brezenski are enclosed for Messrs. Kauffman, Behrend, and Zook.

UNITED STATES DEPARTMENT OF THE INTERIOR
FEDERAL WATER POLLUTION CONTROL ADMINISTRATION
NORTHEAST REGION
HUDSON-DELAWARE BASINS OFFICE
EDISON, NEW JERSEY 08817

October 13, 1970

Mr. Thomas Iezzi
Rohm & Haas Co.
Box 219
Bristol, Pa.

Dear Mr. Iezzi:

Enclosed is a copy of the FWQA recommended methodology for lead, cadmium and arsenic. Since your laboratory has been splitting samples (Tulpehocken Creek) with us for arsenic analysis, it would be desirable to discuss methodology. An invitation is being extended to you for the purpose of visiting our facilities and in the process for discussing analytical techniques for assaying arsenic in water and sediment samples.

Please contact me at your convenience so that we may set a tentative date for this meeting.

FOR THE REGIONAL DIRECTOR:

Sincerely yours,

Francis T. Brezenski
Chief, Laboratory Branch

1-201-548-3347

Enclosure:
FWQA Recommended Methodology
for lead, cadmium & arsenic.

OPTIONAL FORM NO. 10
MAY 1962 EDITION
GSA FPMR (41 CFR) 101-11.6

UNITED STATES GOVERNMENT

Memorandum

FEDERAL WATER QUALITY ADMINISTRATION
DIVISION OF WATER QUALITY RESEARCH
ANALYTICAL QUALITY CONTROL LABORATORY
1014 BROADWAY, CINCINNATI, OHIO 45220

TO : Regional Coordinators

DATE: September 28, 1970

FROM : Director,
Analytical Quality Control Laboratory

SUBJECT: Analytical Methods for Lead, Cadmium and Arsenic

The deep concern of the FWQA over the presence of mercury in surface waters, industrial wastes, sediments and aquatic organisms has initiated a further anxiety about the possible presence of other toxic metals in the aquatic environment. Of the seven metals for which mandatory limits in drinking water have been set (Public Health Service Drinking Water Standards, 1962) lead, cadmium and arsenic appear to be prime candidates for future investigations.

Analytical procedures for arsenic, lead and cadmium are outlined in FWPCA Methods for Chemical Analysis of Water and Wastes, November, 1969) in the section on Metals, pp 87-126. Information relevant to detection limits, sample handling, interferences, concentration techniques, etc., are detailed on pp 87 through 101. Specific information on the measurement by atomic absorption of arsenic appears on pp 104-105, for cadmium, pp 106-108 and for lead, pp 115-116. The atomic absorption procedure for cadmium and lead should be used in FWQA investigations.

Although arsenic can be measured by atomic absorption, the sensitivity achieved is not satisfactory for analysis of routine samples. For this reason FWPCA Methods for Chemical Analysis of Water and Wastes reference The silver diethyl dithiocarbamate (SDDC) colorimetric procedure which appears in Standard Methods for the Examination of Water and Wastewaters, 12th Edition, pp 56-57. The SDDC method is a very sensitive colorimetric procedure, capable of detecting less than 10 µg/l of arsenic. However, the same problem exists for detection of arsenic that also exists for certain other metals, particularly mercury. That is, the arsenic is sometimes organically bound and may not react in the final color formation step unless it is first reduced to an inorganic form. The digestion step detailed in Standard Methods (p 59, paragraph 4.1 under Procedure) appears under Method B, Mercuric Bromide Stain Method and may be overlooked by an analyst using the SDDC procedure.

Studies done several years ago in the AQCL indicate that without digestion, recovery of arsenic from organic arsenicals is less than 20%. Use of the sulfuric-nitric acid digestion improves recovery to about 50% and addition of potassium persulfate to the acid digestate further improves recovery to about 80%. (Table 1, attached). Persulfate is incorporated in the arsenic digestion by adding 1 gram potassium persulfate, dissolved in the 25 ml of distilled water in Standard Methods, 12th Edition, p 59, paragraph 4.1.

Metals or salts of metals such as cobalt, mercury, nickel, silver, palladium, copper, chromium and molybdenum are said to interfere with the evolution of arsine[1]. The last three metals do so when present in large amounts. The AQCL plans to check more thoroughly the degree of interference by these metals and, if necessary, attempt removal by ion exchange methods. Contract studies are also under way to improve the atomic absorption procedure for arsenic.

(1) Liederman, I., Bowen, J. E. and Milner, O. I., Determination of Arsenic in Petroleum Stocks and Catalysts by Evolution of Arsine, Anal. Chem. 31, 2052-2055 (1959).

Dwight G. Ballinger

Dwight G. Ballinger

Attachment

Table 1

Recovery of Organically Bound Arsenic* From Spiked Water Samples Using SDDC Method

Compound	Arsenic Added µg/l	No Oxidation Step µg/l	%	H_2SO_4 + HNO_3 µg/l	%	H_2SO_4 + $K_2S_2O_8$ µg/l	%
p-arsanilic acid	1000	200	20	967	97	853	85
	200	-	-	140	70	132	66
o-arsanilic acid	1000	180	18	866	87	1026	103
	200	-	-	180	90	146	73
1-(0-arsonophenyl azo)-2-naphthol-3, 6-disulfonic acid	1000	160	16	566	57	680	68
	200	-	-	132	66	124	62
phenyl arsene oxide	935	<100	<9.4	353	38	760	81
	187	-	-	112	60	152	87

*assay information on arsenic compounds was not available; recovery data may be higher than shown.